

# A Calibration Target for Far-Infrared Spaceborne Applications

M.C. Gaidis, M.S. Anderson, and D.G. Harding

Jet Propulsion Laboratory, California Institute of Technology, Pasadena, CA 91109

Correspondence: gaidis@merlin.jpl.nasa.gov

## ABSTRACT

We describe progress in the development of a calibration target for use in the EOS-MLS 2.5 THz radiometer on NASA's CHEM-1 spacecraft. Although the intended use is as a stable, isothermal black body load at a frequency of 2.5 THz, the design is suitable for use throughout the far-infrared (FIR). A wedge design is used for the target body to enhance the emissivity to desired levels at 2.5 THz. The body is machined from aluminum, giving the best trade between issues such as cost, thermal conductivity, mass, and strength. The target utilizes a *white* coating to reduce the destabilizing effects of periodic solar illumination. The coating can be made relatively thin to allow accurate temperature measurements of the FIR-absorbing medium. Emissivity of greater than 0.99 is achieved at 2.5 THz, while the solar absorptance is estimated at  $< 0.5$ .

**Keywords:** Calibration target, far-infrared, spaceborne, 2.5 THz, black body, absorber, submillimeter, terahertz, radiometer

## 1. INTRODUCTION

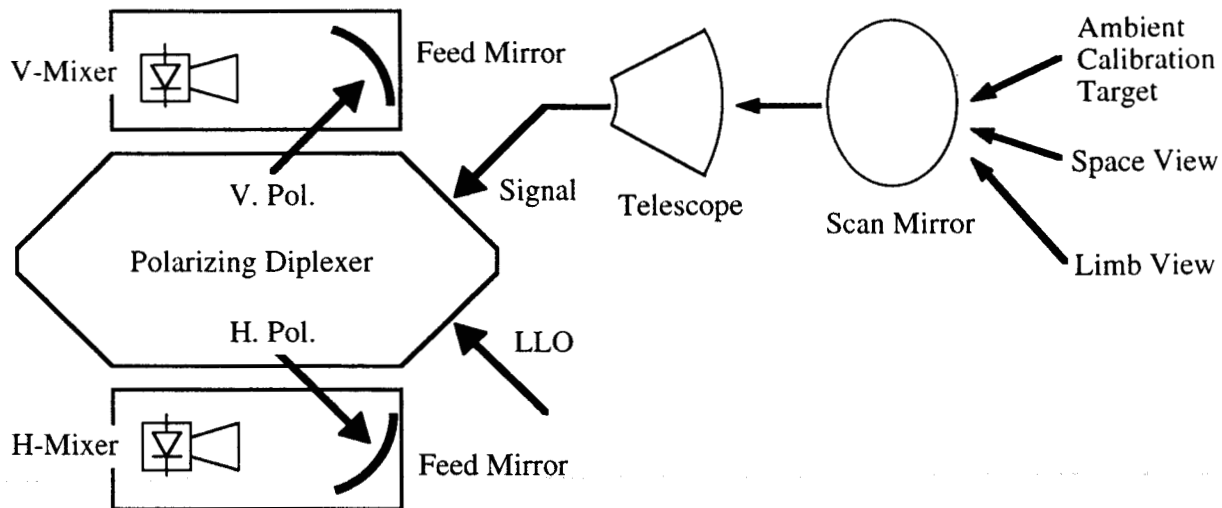
The OH radical plays a significant role in a great many of the known ozone destruction cycles, and has become the focus of an important radiometer development effort for NASA's Earth Observing System (EOS) Chem I satellite, which will monitor and study many tropospheric and stratospheric gases and is scheduled for launch in 2002 [1]. The Microwave Limb Sounder (MLS) instrument on this satellite is the only near-term opportunity to obtain global measurements of this important radical.

The lowest energy OH doublets at 2510 and 2514 GHz fall fortuitously close to a strong methanol laser line at 2522 GHz. A receiver noise of 20,000 K, SSB is expected to provide enough sensitivity for daily global stratospheric maps of OH above 35 km and monthly global maps to 18 km from a limb-sounding satellite in polar orbit. These requirements are consistent with the performance that can be obtained from state-of-the-art room-temperature Schottky diode mixers. The molecular oxygen line at 2502 GHz can also be monitored as a pressure/altitude indicator.

The challenges of producing such a sensitive receiver are numerous. Previous work covers the receiver front end [2]; here we discuss the design, fabrication, and testing of the calibration target. The optical components in the receiver chain are shown in Figure 1. The beam of the dual-polarization receiver is transformed by a telescope to give a beam waist radius of approximately 60 mm at a scan mirror. The scan mirror directs this beam to one of three places: the ambient calibration target (ambient temperature,  $\approx 295$  K), a space view ( $\approx 3$  K), or the limb view (signal from the OH under investigation). The ambient calibration target provides a well defined radiance to calibrate out drifts in receiver performance with temperature fluctuations and aging. This is used in conjunction with the space port view ( $\approx 3$  K) to establish a receiver gain value.

The quality of performance required of the calibration target is dictated by the EOS-MLS desire to have an uncertainty in the absolute value of the measured radiance of less than 3 K in the range 2.520 THz to 2.524 THz. The flow-down of this requirement based on our experimental approach results in the following low-level requirements:

1. Temperature uniformity across the target shall be  $\leq 1.0^\circ\text{C}$
2. Temperature measurement accuracy  $\leq \pm 0.15^\circ\text{C}$
3. Emissivity  $\geq 0.99$
4. Negligible standing waves
5. Suitable for space flight use



**Figure 1:** Schematic diagram of the EOS-MLS 2.5 THz radiometer. LLO refers to a CO<sub>2</sub>-pumped methanol laser local oscillator.

While there are commercially available absorbers which offer quite good emissivity in the 2.5 THz region [3], we find that due to various problems (outgassing, thermal conductivity, structural rigidity...) these did not readily fulfill every one of the five low-level requirements. Below we discuss the present approach which offers what appears to be an acceptable solution.

## 2. TARGET BODY

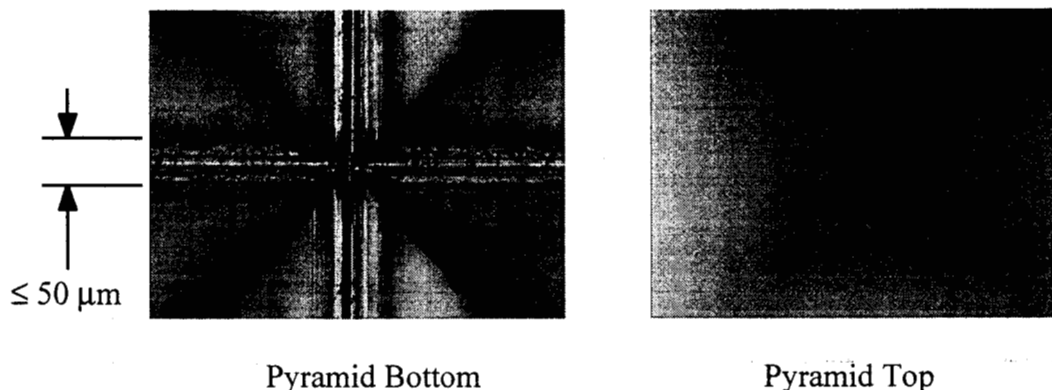
Rather than attempt to develop a frequency-resonant type of absorber [4], we decided on the more brute force but simpler method of a broadband absorber in an absorption-enhancing configuration. The following issues were addressed in designing the structure or body of the target:

1. Pyramids or wedges will be used to increase the number of surfaces off which incident radiation must scatter before back-reflection
2. The tips and bases of the pyramid structures shall be made fine enough to keep direct (zero-order) reflection to allowable values
3. The target size should be large enough to intercept the beam at the  $-40$  dB points to minimize standing wave problems (for the  $\approx 60$  mm beam waist, this requires a target of at least 250 mm in diameter)
4. The target shape must be structurally sound to withstand the stresses of launch vibration, and the target must fit within the THz receiver module
5. Target mass shall be minimized within the above constraints
6. Target lifetime shall be greater than 2 years ground/storage and 5 years in low earth orbit
7. Illumination by the sun at different orbital positions shall not introduce significant temperature changes or gradients
8. Thermometry must be provided with adequate resolution and accuracy, giving a true measure of the effective black body temperature of the target

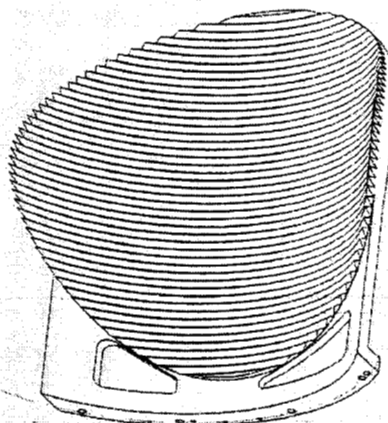
The calibration target was forced to be in a “potato-chip” shape to fit within the THz receiver module and still intercept the beam at the  $-40$  dB point as it reflects from the scan mirror. Because of this loss of symmetry, a wedge profile was chosen over a pyramidal profile, at the expense of some mass increase. Examination of various materials gave us confidence that only a thin layer ( $\approx 200$   $\mu\text{m}$ ) of absorber was needed to achieve required emissivity. Therefore, a relatively high thermal conductivity body could be used, then coated with a thin layer of absorber. Aluminum was chosen for the calibration target body for ease of machining, cost, thermal conductivity, mass, and strength.

The shape of the wedges was a trade-off between mass and performance. One would like a small included angle between wedges to maximize the number of reflections before back-reflection, but the wedge must be significantly larger than a wavelength to ensure predictable behavior. In addition, reasonably-priced machining techniques cannot produce perfectly

sharp tips and valleys, so a larger wedge period is desirable to minimize the overall effects of these “seams.” These considerations are offset by an increase in mass as one tends toward large period and sharp angle. Preliminary experimentation shown in Figure 2 revealed that “seams” of smaller than 50  $\mu\text{m}$  were readily achievable using conventional machining techniques with a specially-ground tool [5].



**Figure 2:** Demonstration of precision machining of pyramid “seams.”



**Figure 3:** Conceptual view of the calibration target. For scale, the circular projected diameter of the target is  $\approx 250$  mm.

The wedge profile was then fixed at 5 mm height and 5 mm period ( $60^\circ$  included angle), resulting in of order 1% of the incident energy being subject to only one bounce from the absorbing surface. The supporting structure adds another  $\approx 7.5$  mm thickness of aluminum behind the wedges, which allows enough thermal conductivity for good temperature uniformity, and provides for straightforward temperature sensor mounting. Figure 3 shows a conceptual view of the calibration target, with its potato-chip shape and mounting bracket. The scan mirror rotates within the target with adequate clearance. Although adding mass, the curved shape greatly enhances structural rigidity, and we have demonstrated the target withstands the EOS-MLS random vibration requirement of 13 g, rms.

Platinum resistance thermometers (PRTs) are buried in the aluminum support, at seven separate places on the rear of the calibration target. Thermal modelling can be used to estimate the effective black-body temperature of the target based on measurements from these seven PRTs. The PRTs are secured with either Stycast 2850FT/24LV [6] or Epo-Tek T7109 [7] high-thermal conductivity electrically-insulating epoxy, the leads are stress-relieved with insulated turret terminals, and the wiring is spot-bonded to clear aluminum surfaces of the target with Stycast 2850FT/24LV. Figure 4 shows the rear of the calibration target and the PRT mounting scheme. Figure 5 shows additional views of the calibration target, including the wedge structures. The white coating is discussed below.

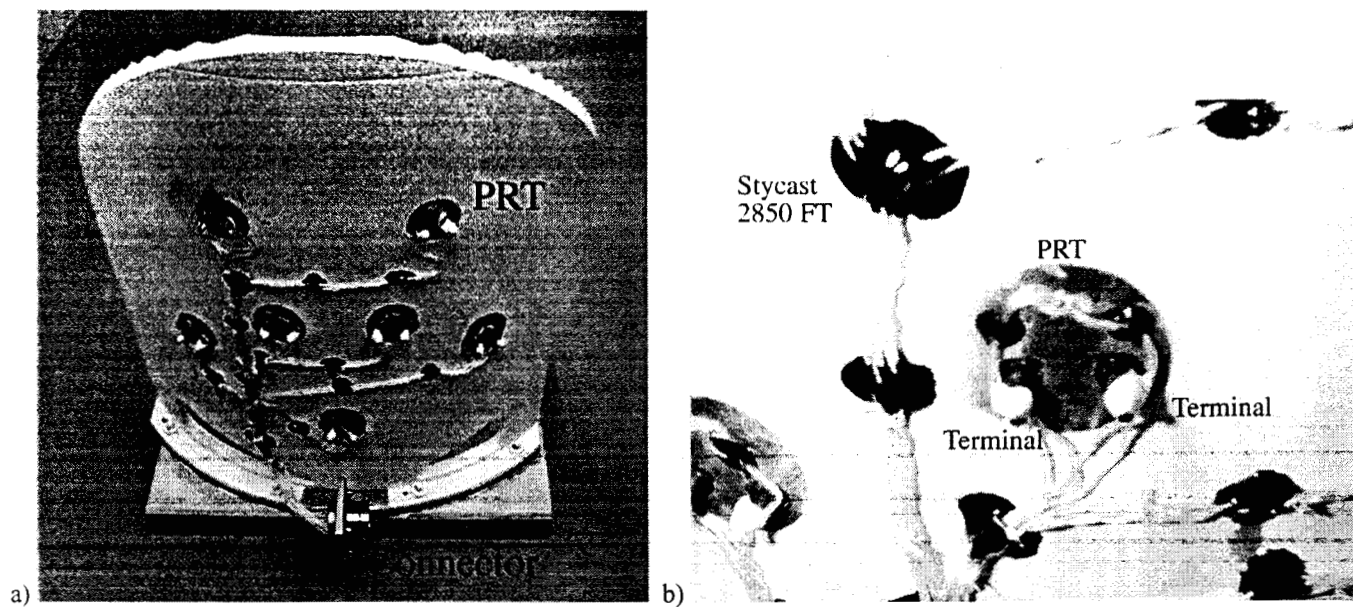


Figure 4: The rear face of the calibration target and the PRT mounting scheme.

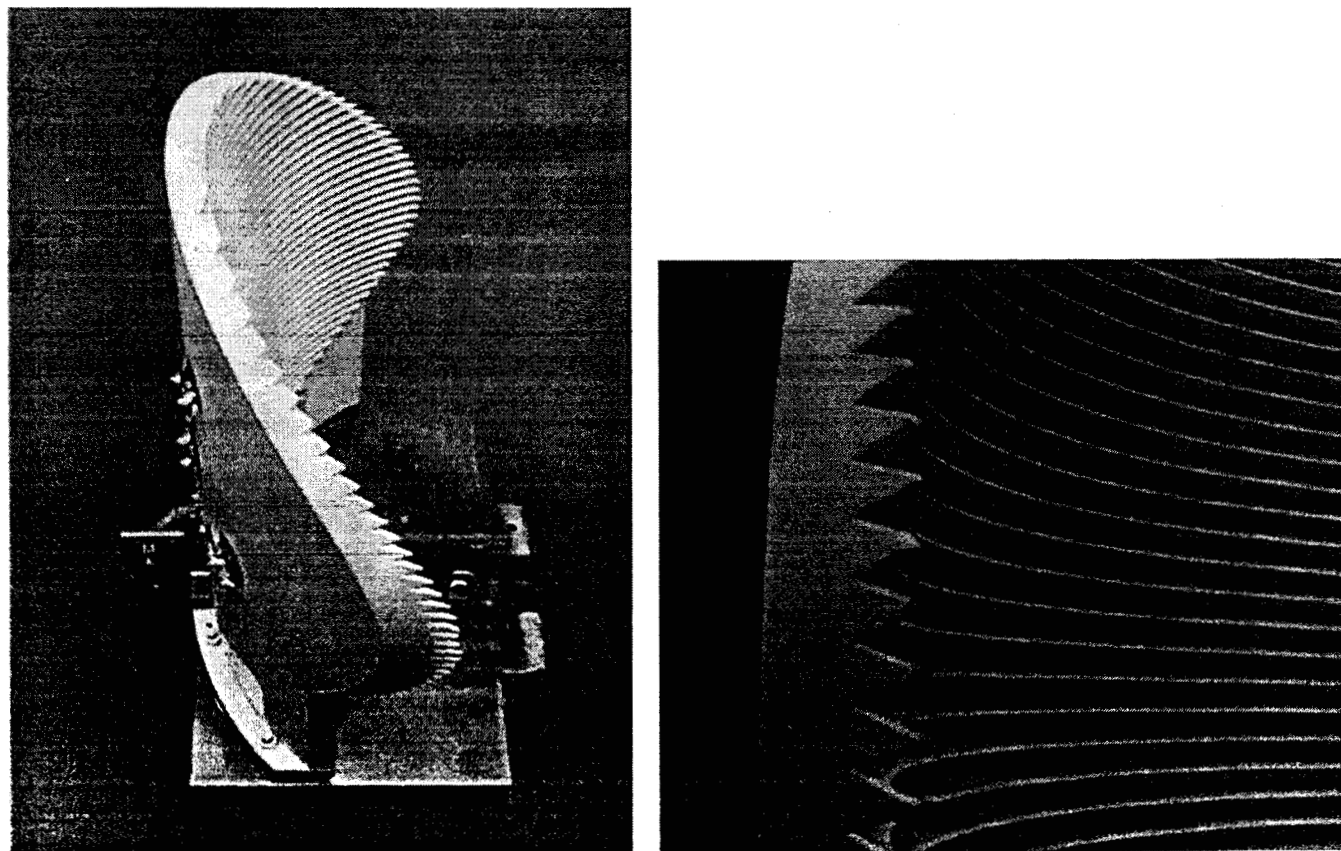


Figure 5: Calibration target side view and wedge closeup view.

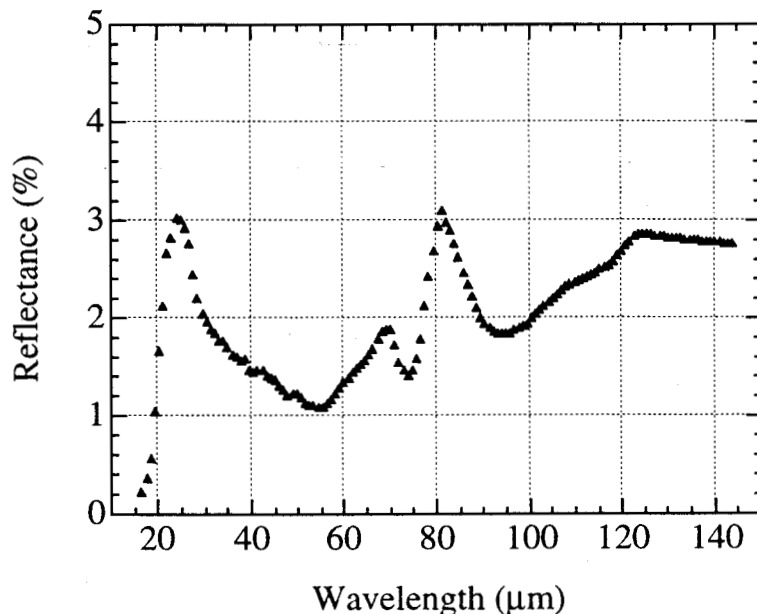
### 3. FIR-ABSORBING COATING

Infrared reflectivity measurements on various absorbing coatings were made using a Bio-Rad FTS 6000 (Cambridge, MA) spectrometer with a wire grid beam splitter and a high temperature ceramic source. The beam is focused to a  $\approx 1$  mm sample spot with an off-axis paraboloid. We use an Analect (Irvine, CA) biconical type diffuse reflectance attachment and a Bio-Rad Deuteride Triglyceride Sulfide (DTGS) detector optimized for the FIR. The specular and diffuse reflected light is collected within a cone  $\approx 22.5^\circ$  from the specular reflected ray. The coating samples were smooth relative to a wavelength, and therefore very little reflected light escapes detection. The measurements were made relative to a specular gold reference target. For our purposes, the gold reference target is assumed perfectly reflecting. The resolution was 8 wavenumbers.

Note that the angle of incidence is  $45^\circ$ , so the effective absorbing film thickness is  $\sqrt{2}$  times the measured film thickness. The  $60^\circ$  wedges on the actual calibration target will similarly add to the path length of a ray through the absorbing material. To avoid confusion, in the following discussion only the measured film thickness is stated.

Initial measurements were made on somewhat obvious choices for coatings, with some history of FIR and submillimeter use [8]. Measurements were concentrated on Nextel Suede [9] and Aeroglaze Z306 with carbon black (Z306/CB) [10], and promising results were found: single-bounce reflectivity of  $\approx 100 - 150 \mu\text{m}$  thickness coatings in the range of 5% for the Nextel, and 10% for the Z306/CB. However, two issues led us to abandon these materials: they absorb strongly in the visible, making the target sensitive to solar illumination, and they outgas with a troublesome opaque deposit (even after a vacuum bakeout was used to fulfill the NASA requirements of total mass loss of  $< 1\%$  and volatile condensable material of  $< 0.1\%$ ).

In an effort to reduce the effects of solar illumination, a white paint, AZ93 [11] was investigated in the FIR. AZ93 is a combination of zinc oxide in a potassium silicate binder. This coating has an extensive space-flight history and surprisingly good longevity – less than 4% deterioration in solar absorptance, and less than 1% change in thermal emittance after more than 700 equivalent solar hours [12]. Quoted values of solar absorptivity and thermal emittance are  $\leq 0.18$  and  $\geq 0.88$ , respectively [11]. To our knowledge, this publication reports the first study of AZ93 in the FIR. Outgassing is acceptable, and adhesion to aluminum is quite good, not worse than 3A by the ASTM D3359A test [11]. The reflectance data shown in Figure 6 reveals that the FIR absorption is quite good as well.



**Figure 6:** Single-bounce reflectance plot of AZ93 with a binder overcoat. Total coating thickness  $\approx 200 - 250 \mu\text{m}$ . The plot is comprised of an average of 6 locations on 3 coated flat aluminum witness plates. The reflectance is negligible between 3 and  $10 \mu\text{m}$ .

Further investigation revealed several more interesting characteristics of the AZ93:

- adhesion is adequate to withstand the 13 g, rms vibration test
- heating to at least 80° C induced no obvious structural degradation
- rapid thermal cycling (dip in liquid nitrogen) to 77 K induces no obvious structural or FIR emissivity changes
- the quality of the AZ93 film appears quite sensitive to application and curing conditions (temperature, humidity, atmosphere...)

The last item above refers to the formation of a chalky film on the surface of the AZ93 during some of our tests (approximately 50% of the tests show such behavior). We have been able to abate this problem by applying a final coating of 10 – 20 µm thick potassium silicate binder, with no obvious problems in the FIR as seen in Figure 6. The humidity and temperature of the application area is tightly controlled, but there is no obvious correlation seen yet with the formation of the chalky film. The AZ93 is applied with a low pressure, high flow spray gun, and, after application to wedges or pyramids, the substrate is turned upside down for curing between coats (this prevents puddling of the low viscosity fluid in the wedge bases, and ensures good coating on the wedge tips). Each coat is ≈ 25 µm thick, and cures for ≈ 30 minutes before the next coat is applied.

To maintain the fidelity of the wedge substrate, it behooves us to minimize the thickness of the absorbing coating. The FIR reflectance has been tested for various coating thicknesses (all without the binder overcoat), and is shown in Figure 7. Best emissivity is found for films at least 200 µm thick, for operation at 2.5 THz (≈ 120 µm wavelength).

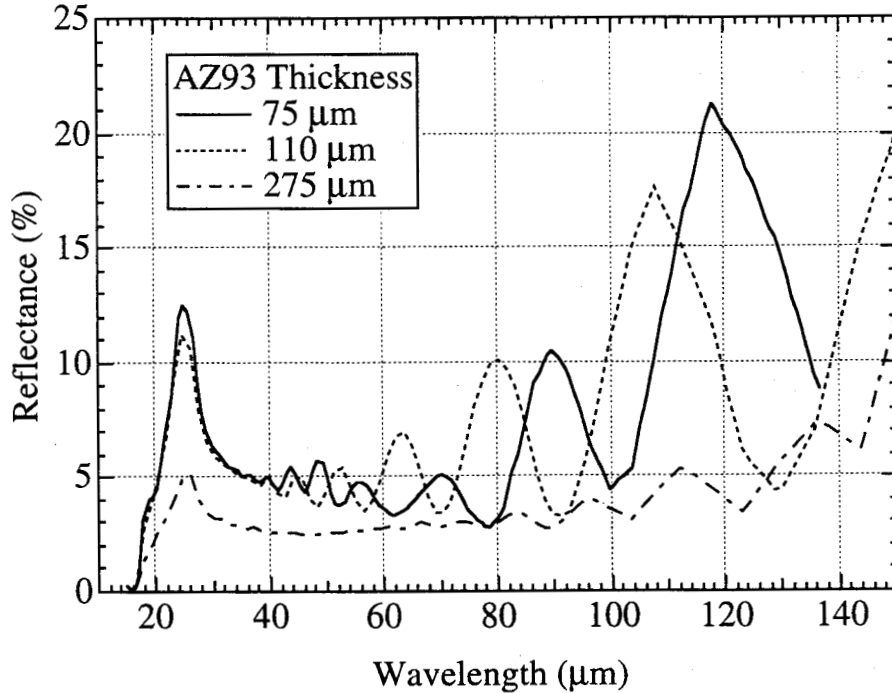


Figure 7: FIR reflectance (single-bounce) vs. thickness of AZ93 coating on flat aluminum plates.

#### 4. DISCUSSION

The FIR reflectance data from the flat aluminum plates can be extrapolated to give a reasonable guess at the reflectance one should expect from a wedged structure, using the following formula:

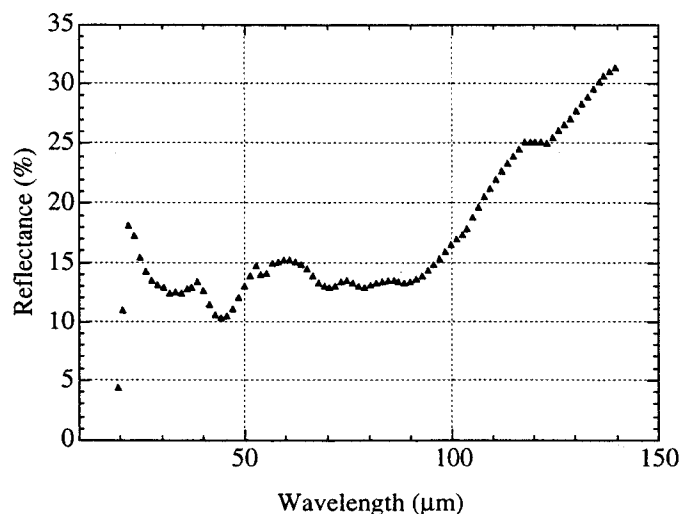
$$R_{\text{total}} \approx R_{\text{flat}}^{(\pi/\theta)},$$

where  $\theta$  is the total included angle of the groove ( $\pi/3$  radians in our case) and  $R_{\text{flat}}$  is the reflectivity of the front surface (e.g. from a flat witness plate) [13]. Based on this formula, one arrives at 2.5 THz emissivity of better than 0.999 for the wedged target described above. Using quoted solar absorptance numbers for AZ93 [11, 12], the same equation implies the wedged structure reflects more than 50% of incident solar power. Coating of surrounding, non-wedged, surfaces with the same AZ93 also assists in reducing the target's thermal fluctuations due to solar illumination. Thus, the target appears to meet the needs of the EOS-MLS 2.5 THz radiometer at this time. Nonetheless, improvements can be made – particularly in the art of coating application. It would be comforting to possess a reliable procedure for obtaining powder-free coatings at will.

One can hypothesize that the powder is zinc oxide and potassium carbonate residue which is released by degradation of the potassium silicate binder. A chemical reaction involving carbon dioxide and moisture can cause such degradation, and can be very sensitive to ambient conditions. If future tests find this to be the case, this implies that long-term integrity of the coating may depend on storage conditions, with best results found using a low humidity storage environment. Additives may be used to improve the robustness of the silicate binder, similar to those used in common window glass, although the effort involved in testing such a broad parameter space is likely not cost-effective.

We do feel, however, that the potassium silicate binder should be the primary subject of further investigations to improve this coating for FIR applications. The silicate is most likely responsible for the FIR absorption while the zinc oxide serves to increase the mid-IR emissivity and the visible reflectance [14] (both useful for maintaining a thermally stable target in the presence of solar illumination). The milling of the zinc oxide pigment with the potassium silicate binder does not appear to induce any chemical reaction between these compounds as evidenced by the white (rather than clear) color of the mixture. Although zinc oxide can dissolve in an alkali solvent, it is not as reactive as silicate, and thus the silicate stays bonded to the potassium during milling/spraying/drying.

We have examined a thin coating of the potassium silicate binder (with no zinc oxide pigment), and find indeed that it is quite effective at absorbing FIR radiation. The results are presented in Figure 8. This is consistent with a silicon-to-oxygen ratio in the AZ93 binder such that the Si-O-Si bonds open to rather large angles (and a correspondingly lower vibrational “spring constant”) and can be excited by relatively low frequency radiation.



**Figure 8:** FIR reflectance of  $\approx 25 \mu\text{m}$  thick potassium silicate binder on a flat aluminum plate.

## 5. CONCLUSION

Certain silicates, particularly when used in a “wedged” geometry, can be effectively used as black-body calibration loads in the FIR region. The high thermal conductance of the rather thin layers needed for FIR absorption allows accurate temperature measurement of the actual absorbing layer. Strong, nearly isothermal support structures can be used to provide a reliable, easily characterized calibration target for space flight applications. The rather simple doping of the silicate with zinc oxide can enhance the films properties in the visible to mid-infrared regions without seriously affecting the FIR performance.

Obvious drawbacks to using such an absorber configuration are that the simple application method (airbrush) of the low-viscosity coating may not preserve the integrity of the wedge shape, and that the use of the electrically-insulating silicate will result in charging of the surface in space-flight use. Presuming the "art" of AZ93 application can be fine-tuned to a reliable process without formation of chalky films, the drawbacks are relatively minor, and this coating holds great promise for use as an absorber in the FIR.

Future work will involve further investigation into the nature and formation of the chalky residue, the use of the silicate binder without zinc oxide pigment, and more precise exploration, control, and monitoring of the ambient conditions during coating application and storage.

## 6. ACKNOWLEDGEMENTS

The research described in this publication was carried out at the Jet Propulsion Laboratory, California Institute of Technology, under a contract with the National Aeronautics and Space Administration. We would like to acknowledge the many valuable contributions to this work by the following people: J. Apodaca, J. Campanella, J. Carson, G. Forsburg, J. Gaidis, R. Jarnot, T. Lespron, R. Mell, S. Park, H. Pickett, P. Siegel, C. Smith, S. Smith, and J. Squires.

## 7. REFERENCES

1. J.W. Waters *et al.*, "The UARS and EOS Microwave Limb Sounder (MLS) Experiments," *J. Atm. Sciences*, **56**, pp. 194 – 218, 1999; H.M. Pickett and D.B. Peterson, "Comparison of Measured Stratospheric OH with Prediction," *J. Geophys. Res.-Atm.*, **101**, pp. 16789 – 16796, 1996.
2. M. C. Gaidis, H.M. Pickett, P.H. Siegel, C.D. Smith, R.P. Smith, and S.C. Martin, "A 2.5 THz Receiver Front-End for Spaceborne Applications," submitted for publication in *IEEE Trans. Micr. Theory Tech.*, January 2000.
3. TK-RAM from Thomas Keating, Ltd., Station Mills, Billingshurst, West Sussex, RH14 9SH, England (<http://qmciworks.ph.qmw.ac.uk/homepage.htm>); FIRAM-500 and TERASORB from University of Massachusetts Lowell, Submillimeter-Wave Technology Laboratory, 175 Cabot Street, Lowell, MA 01854 (<http://stl.uml.edu>).
4. R.H. Giles, A.J. Gatesman, J. Fitzgerald, S. Fisk, and J. Waldman, "Tailoring Artificial Dielectric Materials at Terahertz Frequencies," in the *Proc. 4<sup>th</sup> Intl. Symp. Space THz Tech.*, Los Angeles, CA, April 1993.
5. Thomas Keating, Ltd., Station Mills, Billingshurst, West Sussex, RH14 9SH, England
6. Stycast 2850FT/24LV from Emerson Cuming, 28 York Avenue, Randolph, MA 02368
7. Epo-Tek T7109 from Epoxy Technology, 14 Fortune Drive, Billerica, MA 01821
8. S.M. Smith and R.V. Howitt, "Survey of Material for an Infrared-Opaque Coating," NASA Technical Memorandum 88204, February 1986; J.R. Grammer, L.J. Bailin, M.D. Blue, S. Perkowitz, "Absorbing Coatings for the Far Infrared," *SPIE Vol. 257 Radiation Scattering in Optical Systems*, pp. 192 – 195, 1980.
9. 2-part absorptive Nextel Suede coating, p/n 3101-C10 with primer 911-P5 and airbrush component SV3148, from Red Spot Paint and Varnish, P.O. Box 418, Evansville, IN 47703-0418
10. Aeroglaze Z306 Flat Black Absorptive Polyurethane, 9929 Primer, and 9958 Thinner, from Lord Industrial Coatings, 2000 West Grandview Blvd., P.O. Box 10038, Erie, PA 16514-0038; Carbon Black type "Raven 2000" from Columbian Chemicals Company, 1600 Panowood Circle, Atlanta, GA 30339; Z306:CB tested in weight ratios of 2:1, 2.7:1, 6.7:1, and 8:1.
11. AZ93 White Thermal Control Coating, AZ Technology, 4901 Corporate Drive, Suite 101, Huntsville, AB 35805
12. D.L. Edwards *et al.*, "Radiation Induced Degradation of the White Thermal Control Paints Z-93 and Z-93P," NASA Technical Memorandum 108518, October 1996; Also, personal communication with Richard Mell of AZ Technology regarding Long Duration Exposure Facility (LDEF) data..
13. S. Janz, D.A. Boyd, and R.F. Ellis, "Reflectance Characteristics in the Submillimeter and Millimeter Wavelength Region of a Vacuum Compatible Absorber," *Int. J. Infrared and Millimeter Waves*, **8**, pp. 627 – 635, 1987.
14. R.A. Nyquist and R.O. Kagel, *Infrared Spectra of Inorganic Compounds*, New York: Academic Press, 1971; P.T.T. Wong and E. Whalley, "Infrared and Raman Spectra of Glasses," *Discuss. Faraday Soc.*, **50**, pp. 94 – 102, 1970.